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COC. 1



No. 8103258

By virtue of a direction given under Section ³⁰~~17~~(1) of the Patents Act, ¹⁹⁷⁷~~1949~~, the
application is proceeding in the name of **COAL INDUSTRY (PATENTS) LIMITED** of **Hobart House,**
Grosvenor Place, London SW1X 7AE.

3 FEB 1981

The Comptroller
The Patent Office
25 Southampton Buildings
London WC2A 1AY

3981 4263 PAT 000600

03258

REQUEST FOR THE GRANT OF A PATENT

THE GRANT OF A PATENT IS REQUESTED BY THE UNDERSIGNED ON THE BASIS OF THE PRESENT APPLICATION

I Applicant's or Agent's Reference (Please insert if available) GJE/181/7. GKJ/JDT

II Title of Invention
Gas sensor

III Applicant or Applicants (See note 2)

Name (First or only applicant) City Technology Limited

Address The City University, Northampton Square, London EC1V 0HB

Nationality a British Company

Name (of second applicant, if more than one)

Address

Nationality

IV Inventor (See note 3)

(a) The applicant is not the inventor or

(b) A statement on Patents Form No. 7/77 is/will be furnished

V Authorisation of Agent (See note 4)

~~XXXXXXXXXXXXXXXXXXXX~~

General Authorisation
GILL JENNINGS & EVERY

VI Address for Service (See note 5) Room 21/77.

53/64 Chancery Lane
London WC2A 1HN

VII Declaration of Priority (See note 6)

Country

Filing date

File number

VIII The Application claims an earlier date under Section 8(3), 12(6), 15(4) or 37(4) (See note 7)

Earlier application or patent number and filing date

☒ Check List (To be filled in by applicant or agent)

A The application contains the following number of sheet(s)

1 Request 1 1/77 Sheet(s)

2 Description 18 Sheet(s)

3 Claim(s) - Sheet(s)

4 Drawing(s) 3 x 2 Informal Sheet(s)

5 Abstract - Sheet(s)

B The application as filed is accompanied by:-

1 Priority document no

2 Translation of priority document no

3 Request for Search no

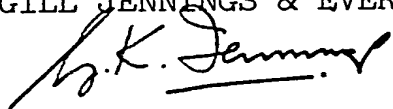
4 Statement of Inventorship and Right to Apply no

5 Separate Authorisation of no

☒ It is suggested that Figure No of the drawings (if any) should accompany the abstract when published

☒ Signature (See note 8)

Agents for the Applicants (By General Authorisation)
GILL JENNINGS & EVERY



G. K. Jennings

NOTES:

1 This form, when completed, should be brought or sent to the Patent Office together with the prescribed fee and two copies of the description of the invention.

2 The name, address and nationality of each applicant are to be stated in the spaces provided at III. Names of natural persons should be indicated in full. Bodies corporate should be designated by their corporate name. If there are more than two applicants the information concerning the third (and further) applicants should be given on a separate sheet.

3 Where the applicant or applicants is/are the sole inventor or the joint inventors, the declaration (a) to that effect at IV should be completed and the alternative statement (b) deleted. If however this is not the case the declaration (a) should be struck out and a statement will then be required to be filed upon Patents Form No.7/77.

4 If the applicant wishes to appoint an agent, his name and address of his place of business shall be indicated in the spaces available at V and VI; such indication will be considered to be an authorisation for the agent to prosecute the application up to grant of a patent and to service any patent so granted.

5 If no authorised agent is appointed an address for service in the United Kingdom to which all documents and notices may be sent must be stated at VI. It is recommended that a telephone number be provided if available.

6 The declaration of priority at VII should state the date of the previous filing and the country in which it was made and indicate the file number, if available.

7 When an application is made by virtue of section 8(3), 12(6), 15(4) or 37(4) the appropriate section should be identified at VIII and the number of the earlier application or any patent granted thereon identified.

8 An agent may sign only when previously authorised. An express authorisation signed by the applicant(s) must be received by the Patent Office before the expiry of 3 months from the filing date.

9 Attention of applicants is drawn to the desirability of avoiding publication of inventions relating to any article, material or device intended or adapted for use in war (Official Secrets Acts, 1911 and 1920). In addition after an application for a patent has been filed at the Patent Office the comptroller will consider whether publication or communication of the invention should be prohibited or restricted under section 22 of the Act and will inform the applicant if such prohibition is necessary.

10 Applicants resident in the United Kingdom are also reminded that, under the provisions of section 23 applications may not be filed abroad without written permission or unless an application has been filed not less than six weeks previously in the United Kingdom for a patent for the same invention and no direction prohibiting publication or communication has been given or any such direction has been received.

City Technology Limited

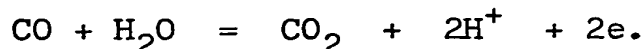
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Gas sensor

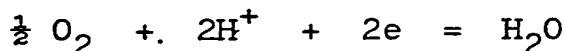
This invention relates to electro-chemical gas sensors in which the gas or vapour to be sensed is caused to react at one electrode of an electro-chemical cell which also includes a counter electrode

5. and an intervening body of electrolyte and may also include a third reference electrode, in such a way that the current through the cell, or a characteristic potential of the cell, is a function of the concentration of the gas or vapour to be sensed.
10. The invention is particularly applicable to the sensing of oxidisable gases and vapours such as carbon monoxide, hydrogen sulphide, ethyl alcohol, sulphur dioxide, nitric oxide and so forth.

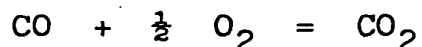
- The principles will be described using
15. the sensing of carbon monoxide as an example but it is understood that this is simply one example of its applicability to a wide range of gases. Thus, at one electrode (anode) of the sensor carbon monoxide is electrochemically oxidised as illustrated
20. by the equation



At the counter electrode a reduction process must take place, for example the reduction of oxygen



25. The overall sensor cell reaction is the sum of these two electrode reactions, namely



By Faraday's law the flux of carbon monoxide reacting at the anode is proportional to the current.

5. If the flux of carbon monoxide to the anode is highly restricted by a suitable diffusion barrier, then substantially all the carbon monoxide reaching the anode can react, thus reducing its concentration at this point to essentially zero, so that the flux of carbon monoxide is determined
10. by the diffusion resistance of the diffusion barrier and the concentration of carbon monoxide outside the diffusion barrier. There is therefore a direct link between the concentration of carbon monoxide and the current delivered by the sensing
15. cell.

- The diffusion barrier may take the form of a thin non-porous plastic film through which the gas to be sensed permeates by a process of solution diffusion. This, however, results in
20. a sensor with a very high temperature co-efficient and a more preferable barrier to use is the gaseous diffusion barrier described in our U S patent no: 4,132,616 which results in a low temperature co-efficient and excellent stability.

25. To promote the reaction of the gas to be sensed, for example carbon monoxide, at the sensing electrode, the electrode needs to contain a suitable catalyst preferably in high surface area form. Noble metals such as platinum, gold, palladium,
30. their mixtures or alloys are commonly used sometimes with other additions to help promote the reaction. The chosen electrode material must also be a reasonable electronic conductor.

- To allow the gas to reach the electrode catalyst without electrolyte being able to leak out of the cell through the same permeation path, a thin non-porous plastics film may be used, but
5. a preferred form of electrode is the so-called hydrophobic electrode, such as used in fuel cell technology. In this type of electrode the finely divided active electrode is intimately mixed with fine particles of polytetrafluorethylene (PTFE)
10. which act as a binder and which, being hydrophobic, are not wetted by aqueous electrolytes and so maintain paths for gas permeation throughout the electrode. This catalyst mix may be contained in a suitable conducting mesh, which is then finally
15. "waterproofed" with a layer of porous PTFE on the gas side.

- When the gas to be sensed is a reducing agent such as carbon monoxide or hydrogen sulphide which is oxidised at the sensing electrode (anode),
20. the counter electrode (cathode) must be capable of sustaining a reduction (cathodic) process. Examples of such electrodes are lead dioxide electrodes and oxygen reduction electrodes. A third, reference electrode, may also be included to monitor or control
25. the potential of the sensing electrode.

- An acid electrolyte is normally chosen, since with alkaline electrolytes there are problems with carbon dioxide interference when monitoring gases in ambient air or in flue gases. The acid
30. used may, for example, be sulphuric acid, phosphoric acid, perchloric acid.

Clearly all the components of the sensor

that will be in contact with the electrolyte such as electrodes, current collectors and the sensor cell housing must be of materials compatible with the electrolyte. For this reason plastics such as acrylics, polyethylene, polypropylene, PTFE, ABS, have been used for constructing cell housings.

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There is an increasing demand for gas sensing instruments particularly for safety monitoring, pollution control, fire detection and flue gas analysis. There is a special demand for portable, relatively inexpensive instruments and for these to fit the application requirements, compact, lightweight, robust, reliable and relatively inexpensive sensors are needed.

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A particular problem which arises in instruments of this type is to ensure an adequate electrolyte connection between the electrodes, without which the basic operation of the cell is adversely affected. This connection needs to be stably maintained regardless of attitude changes and of the appreciable volume changes in the electrolyte that can result from changes in ambient humidity.

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It is an objective of this invention to provide a small compact electrochemical gas sensor that is simple in construction and has a high degree of integrity, reliability and freedom from the proneness to leakage and the problems mentioned above that have often been found with electrochemical sensors.

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According to the present invention a gas sensor is in the form of an electrochemical cell having an anode comprising catalyst mixed with PTFE particles and pressure bonded to porous unsintered PTFE tape and a counter electrode spaced

- from the anode by a hydrophilic, non-conducting porous separator having an extension of similar material which constitutes a wick passing through an opening in the counter electrode into an
5. electrolyte expansion chamber which is partially filled with electrolyte, the combination of anode, counter electrode, separator/wick and electrolyte expansion chamber forming a sealed assembly and having provision for access to the anode for gas
10. to be detected and connections from the electrodes to external terminals.

- The provision of the wick with the particular feature that it connects to the electrolyte reservoir through an opening in the cathode, which may be
15. formed by a hole or slit in the cathode, ensures an adequate supply of electrolyte to the space between the electrodes quite independently of any volume changes of the electrolyte or the attitude of the cell and permits very close spacing of the electrodes
20. with consequent reduction in size of the cell and hence the sensor as a whole.

- A further advantage of the construction is excellent sealing and freedom from leaks which derives from the use of unsintered PTFE tape at
25. the sealing areas. This is particularly effective not only because the PTFE is hydrophobic, but also because it is composed of very small spheres and/or fibrils, the radius of curvature of which highly increases the resistance to aqueous penetration for
30. contact angle reasons, this property of PTFE tape being, of course, well known. A cell in accordance with the invention is found to be particularly suitable for the measurement of carbon monoxide, hydrogen sulphide and alcohol.

- The counter electrode preferably comprises an oxygen reduction electrode of similar construction to the anode, i.e. comprising catalyst mixed with PTFE particles and bonded to a porous PTFE tape, with its supply of oxygen being obtained from the ambient air by radial diffusion inwardly through the porous PTFE tape from the perimeter of the tape. As with previous cells of this kind, a third, oxygen reduction, reference electrode may be included, this being of the same type as the oxygen reduction electrode described above and receiving its oxygen supply in similar manner and being most conveniently provided on the same PTFE tape which carries the oxygen reduction counter electrode but having a separate electrical connection. This may be used to monitor or control the potential of the sensing electrodes, as previously, or for novel methods of operation to be described later.
- With any of the arrangements described above a diffusion barrier may be included to restrict the access of gas to the anode, this diffusion barrier being preferably of the gas phase diffusion type which can take the form of a simple capillary restriction.

Examples of construction embodying the principles of this invention are illustrated in the accompanying drawings, in which:-

- Figure 1 is an exploded longitudinal section;

Figures 1A and 1B are plan views of top and bottom plates respectively;

Figure 2 is a plan view showing the arrangement of the electrodes and current collector; and

Figure 3 is a circuit diagram.

5. These drawings are not to scale, the thicknesses of some of the components, for example PTFE tape and current collectors being emphasised for the sake of clarity.

10. Referring to Figure 1, an anode 1 comprises porous PTFE tape to which is bonded a catalyst/PTFE mixture, the latter covering an area of smaller diameter than the tape. A cathode 2 is of similar construction but with a hole 3 through which a wick 4 passes to an electrolyte reservoir/expansion chamber 5. Current collectors 6, 7 contact the anode and cathode respectively (see also Figure 2) and lead out to terminal posts 8 (one only shown).
15. The wick 4 extends from a separator 9 which with a further separator 10 and an annular gasket 11, which may conveniently be cut from porous PTFE tape, makes up an interior sandwich between the electrodes.
20.

Above the anode 1 is an O-ring 12 with a rigid retaining ring 13 of smaller height than the O-ring, the underside of which has slots (not shown) to allow egress of the current collectors 6 and 7. A top plate 14 carries the capillary holes 15 which form the diffusion barrier for restricting access of the gas to be detected and has a cavity 16 to allow for diffusion across the anode. A bottom plate 17 carries the electrolyte reservoir/expansion chamber 5, the wick 14 passing through a hole 18. In the plan views of Figures 1A and 1B the holes 19 are for the terminal posts 8, the other holes being for assembly bolts.
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- After assembly the whole is clamped together with bolts and nuts (not shown) through the top and bottom plates 14 and 17. The nuts and bolts are tightened sufficiently to suitably compress the O-ring. The outer annular portions of the PTFE tape in the electrodes 1 and 2, not covered with catalyst, are thus brought into intimate compressive contact with the PTFE gasket 11. The PTFE moulds around the current collectors leading out of the cell from the electrodes. Sealing of very high integrity is thus achieved. The height of the ring 13 is chosen, so that the desired degree of compression is obtained by simply bolting up tight to this. O-ring compressions of between 15% and 50% have been successfully employed.
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- With the assembly upside down electrolyte may now be introduced into the reservoir 5 and the cover plate 20 sealed in position in the plate 17. The electrolyte wets up the separators 9 and 10 via the wick 4 to form the electrolyte connection between the electrodes. The amount of electrolyte is chosen to only partially fill the reservoir/ expansion chamber 5 and so that volume changes resulting from gain or loss of water during the operation of the sensor may be accommodated.
20. plate 17. The electrolyte wets up the separators 9 and 10 via the wick 4 to form the electrolyte connection between the electrodes. The amount of electrolyte is chosen to only partially fill the reservoir/ expansion chamber 5 and so that volume changes resulting from gain or loss of water during the operation of the sensor may be accommodated.
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- In Figure 2 the shaded areas represent the PTFE bonded catalyst areas, (a) showing the anode 1 and (b) the cathode 2. (c) Shows the arrangement for a three electrode system, one segment acting as cathode and the other as reference
30. anode 1 and (b) the cathode 2. (c) Shows the arrangement for a three electrode system, one segment acting as cathode and the other as reference

electrode. Although shown as of equal area the relative areas of the cathode and reference electrode may be varied as desired.

5. Ingress of oxygen to the perimeter of the cathode occurs via the clearance between the current collectors 6, 7 and the slots in the O-ring retaining ring 13. To ensure good distribution around the perimeter a narrow, shallow annular channel may be cut in the bottom plate 17 with an outside diameter the same as the inside diameter of 13. From the perimeter the oxygen diffuses radially inwardly through the porous PTFE tape of the cathode 2 to reach the cathode catalyst, the PTFE tape retaining sufficient diffusibility for this purpose even when compressed by the O-ring 12. The diffusibility requirements are discussed in more detail later. If a third, oxygen reduction reference electrode is fitted, then it receives its oxygen supply by a similar route.
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- In an alternative arrangement the O-ring 12 is placed in between the electrodes 1 and 2 and the gasket 11 is omitted. The arrangement shown in Figure 1 is, however, preferred since it gives more reliable sealing and avoids contact between the electrolyte and the O-ring.
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- Suitable materials for a number of the components were discussed earlier. The current collectors 6 and 7 must be a suitably inert conducting material and platinum ribbon is very suitable. The wick and separator material must also be inert to the electrolyte. Borc silicate
- 30.

glass fibre filter mat or polyester fibre mat are suitable materials.

The electrolyte concentration is preferably chosen to be approximately in equilibrium with the mean humidity that will be encountered in use to minimise overall volume changes due to loss or pick up of water. For example 10-N sulphuric acid or 50% phosphoric acid is suitable for mean relative humidities of about 65%.

Although the assembly has been described as bolted together other methods may also be used. For example glueing, ultra-sonic welding, heat welding or spring clips may be used in place of bolts. Alternatively the assembly may be enclosed in an outer metal tube and, after suitable compression of the O-ring, the ends of the metal tube crimped over to secure and maintain the assembly under pressure. In this arrangement the bolt holes through 14 and 17 are obviously unnecessary and the current collectors 6 and 7 are led out through the holes 19. For a three electrode system Figure (c), an additional hole will be necessary.

In a further embodiment of the invention the capillaries are formed using plastic sleeves in enlarged holes 15, the sleeve material being chosen to have a larger co-efficient of thermal expansion than the material of which 14 is made. An increase in temperature will then narrow the bore of the resulting capillary as a result of differential thermal expansion and by suitable choice of materials and dimensions the intrinsically low temperature co-efficient of the gas phase diffusion barrier sensor may be further reduced

to close to zero. The sensitivity of the sensor may be readily varied by varying the number and size of the capillaries to suit any required measurement range.

5. The sensors as so far described are suitable for operating in the diffusion mode. Alternatively, a suitable housing may be fitted to the top plate 14 to allow a gas stream to pass in a continuous flow across the sensor.
10. When used in the diffusion mode it is clear that the ingress of the gas being monitored to the cathode must be much less than the ingress to the anode, so that any reaction of the gas at the cathode will not significantly affect the signal.
15. At the same time the ingress of oxygen to the oxygen reduction cathode must be sufficient to maintain the oxygen reduction reaction.

The situation may be analysed as follows:-

20. If the sensor signal is $=fx \mu A$, where x is the concentration of carbon monoxide in ppm, and f is the sensitivity in μA per ppm, then because the sensor is diffusion limited, the signal can be taken as a measure of gas flux to the anode.

25. The flux of carbon monoxide to the cathode needs to be very much less than that to the anode, say by a factor, b , and the first condition therefore is:

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$$\text{Flux of CO to cathode} \leq \frac{fx}{b}$$

The flux of oxygen to the cathode will be proportionately larger by the ratio of concentrations, assuming that the diffusion constants are the same

for oxygen and carbon monoxide. An extra factor of 2 must also be included since units of current are being used as a measure of flux and one mole of oxygen is electrochemically equivalent to 2 mols of carbon monoxide. So flux of oxygen to cathode = $\frac{fx.2}{b} \frac{2.1 \times 10^5}{x}$

from ambient air with 21% (2.1×10^5 ppm) oxygen.

The second condition is that this flux should be more than adequate to sustain the sensor current (fx), say by a factor a. This can therefore be written

$$\frac{fx}{b} \frac{4.2 \times 10^5}{x} \geq a \text{ fx}$$

or $x \leq \frac{4.2 \times 10^5}{ab}$

For carbon monoxide-interference at the cathode to be insignificant the value of b needs to be of the order of 50 to 100. In the limit there will be an insufficient supply of oxygen when a equals 1, so the limits of carbon monoxide concentration will be 8,400 ppm for b = 50 and 4,200 ppm for b = 100. It can be seen that there is ample leeway for most monitoring applications where the concentrations of interest are much lower than these limits.

A sensor in accordance with the present invention is eminently suitable for meeting this requirement, it being found in practice that the fluxes fall naturally into the required range with the use of readily available porous PTFE tapes for the cathode. Additional control can be easily achieved if necessary by the choice of tape

thickness, porosity, overall diameter and/or the degree of O-ring compression.

5. When the sensor is used in the flow mode, where the gas sample is seen only by the anode, then it is only necessary to ensure that the flux of oxygen to the cathode is sufficient for the sensor current.

10. Many sensors according to the present invention have been made and tested. The majority of these have been approximately 40 mm outside diameter and 15 mm thick, illustrating the small size and compactness made possible. They have shown an excellent record as regards orientation stability, freedom from leakage, low temperature
15. co-efficient and signal stability over long periods of time in tests having so far extended up to seventeen months.

20. When comprising only two electrodes, the cell may be operated with a simple load resistor connected between the anode and cathode and the voltage generated across this by the signal current suitably amplified. Alternatively a current follower or current-voltage converter type of circuit may be used. The provision of a third
25. electrode may be used for conventional potentiostatic control in which the sensing electrode is held at a fixed potential independent of the concentration of the gas to be detected. It may also be used for either of the two novel methods of operation which
30. will now be described.

A problem encountered with simple two electrode sensors employing an air reduction cathode when operating in pure air is that of a residual base line consequent on differences in

- oxygen reduction activity between the anode and cathode. According to a quite separate feature of the invention such residual base lines are biased out by the inclusion of a third electrode in the system and by passing a constant galvanostatic current between this third electrode and the counter electrode, which is normally the cathode. The necessary value of this galvanostatic current required to offset the base line can readily be selected with any particular sensor by trial and error. For preference the cathode is selected to have a higher oxygen reduction activity than the anode, which will show up as a higher open circuit potential in pure air and in this case a positive base line (anodic at the anode) will result. In such a case the galvanostatic current necessary to reduce or eliminate the base line will be a cathodic current to the cathode from the third electrode. As well as the benefit of a reduced baseline it is found that a number of other benefits ensue, notably a much sharper response and greater stability during extended exposures of the sensor to the test gas. The galvanostatic current and its generating circuitry are quite independent of the sensor signal and its circuitry and may be applied to sensors other than those embodying the main features of the invention.

- According to a further feature of the invention the three electrode embodiment of the sensor is connected to an electronic circuit such as that shown in Figure 3 which is so designed that the potential of the sensing electrode, e.g.

the anode when sensing a reducing gas, varies with the concentration of the gas being monitored and the said change in anode potential may then be used as a measure of the concentration of the gas being sensed.

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In Figure 3 the sensing cell is shown within the dotted circle, containing the anode 1, the cathode 2 and the reference electrode 21. A reference voltage rail 22 may be supplied by electronic means or by a suitable battery such as a mercury-zinc cell. A selected voltage to apply between the reference electrode 21 and the point 23 may be achieved by the variable resistance 24. The operational amplifier 25 will then hold this voltage constant. The value of this voltage should be chosen so that undesirable side reactions in the sensing cell are minimised and will normally be chosen so that the anode is in the region of its rest potential in pure air.

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If the anode is now exposed to an atmosphere containing a reducing gas to be monitored e.g. carbon monoxide, the potential of the anode will change and a current will be generated as described earlier. It can easily be seen that the change in anode potential ΔV will be given by $\Delta V = iR$ where i is the current flowing through the sensor between anode and cathode and R is the value of the load resistor 26. The change in anode potential will therefore be a function of the concentration

of carbon monoxide and this signal ΔV may be taken off from points 23, 27 and if necessary amplified for display or other purposes.

5. It would also be possible to use the operational amplifier 25 for amplifying the signal by interposing a gain resistor in the feedback loop 28 with the amplified signal being taken across the gain resistor.

10. It is found that this method of operation, with a load resistor and the anode potential varying as a function of the concentration of the gas being sensed has significant practical advantages compared to the conventional method of potentiostatic operation where the sensing electrode is held at a fixed potential; notably
15. a marked reduction in noise susceptibility.

The following are some examples of sensor in accordance with the invention:-

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EXAMPLE I

- A sensor was constructed according to Figure 1 in which the housing components 13, 14 and 17 were of acrylic plastic; the anode was a mixture of platinum black and PTFE particles pressure bonded to porous PTFE tape 0.1 mm thick of porosity about 25% manufactured by Dodge Industries; the cathode was of similar construction but with a 3 mm diameter hole through its middle; the separator and wick were of Whatman GF/C glass fibre paper; the current collectors were of platinum ribbon; the electrolyte was 10N sulphuric acid, the reservoir of 3 cm³ being approximately one third filled, five capillaries in the top plate were formed by inserts of silicone rubber tubing of wall thickness 1.5 mm and giving capillary dimensions of 1 mm inside diameter and length 4 mm. This sensor had a sensitivity to carbon monoxide of 0.11 μ A/ppm, and a temperature co-efficient of less than 0.05% of signal per °C.

EXAMPLE II

- A sensor was constructed as in Example I but with only a single central control capillary of similar construction and dimensions. This sensor had a sensitivity to carbon monoxide of 0.022 μ A/ppm over the range 0-1000 ppm.

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EXAMPLE III

A sensor was constructed as in Example I, but using finely divided gold as the anode catalyst in place of platinum black, a mixture of platinum black and finely divided gold as the cathode catalyst, and 50% phosphoric acid in place of 10N sulphuric acid. When tested with a mixture of hydrogen sulphide and air the sensor showed a sensitivity of $0.39 \mu\text{A/ppm}$.

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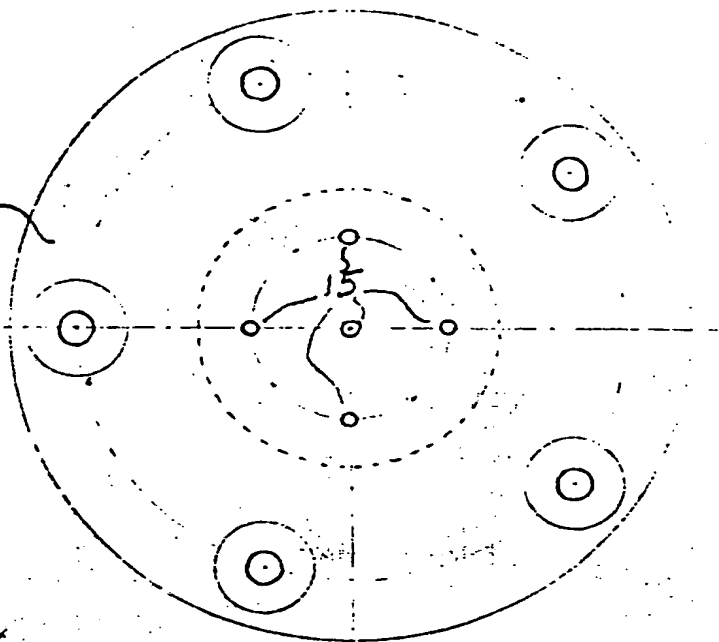
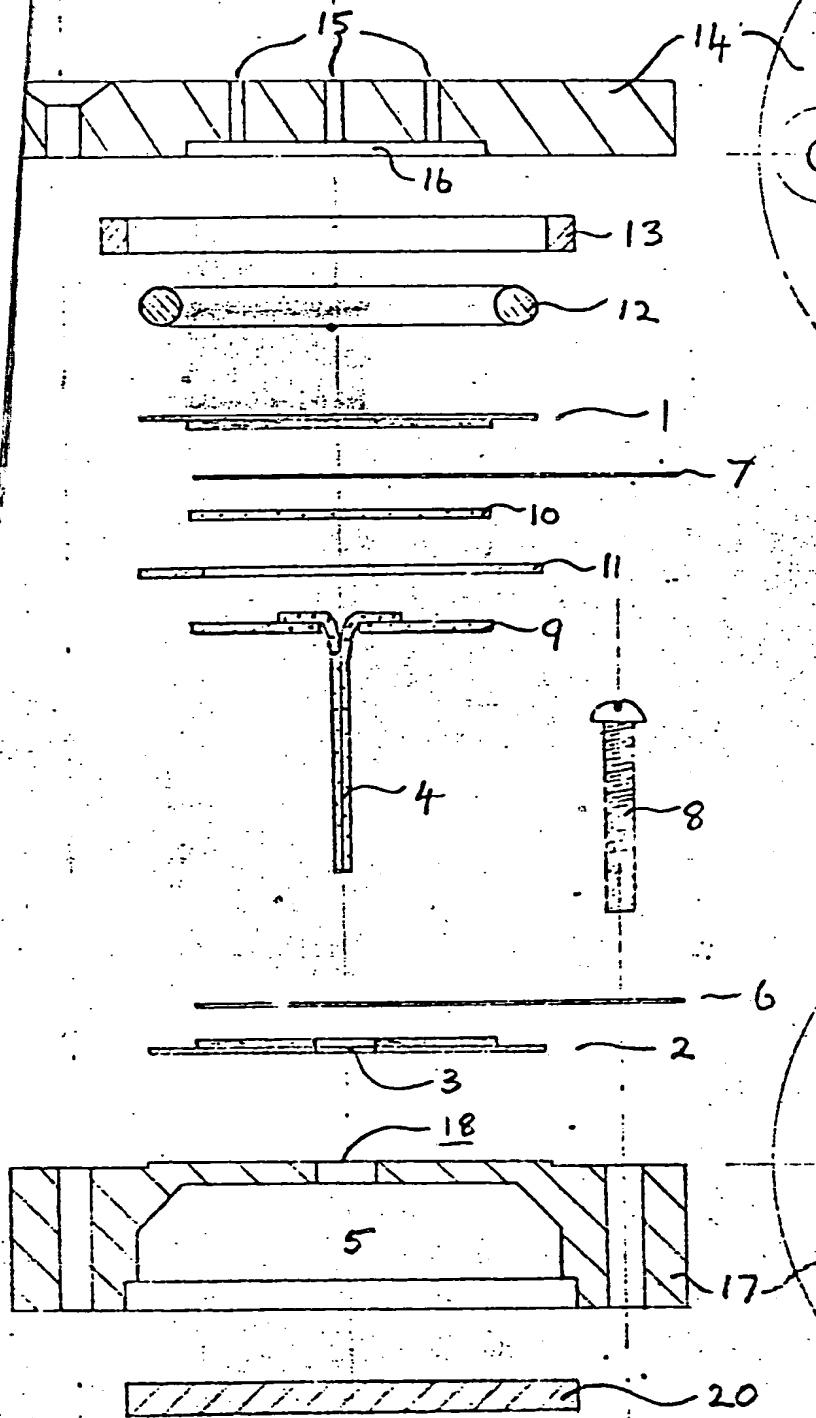


FIG 1A

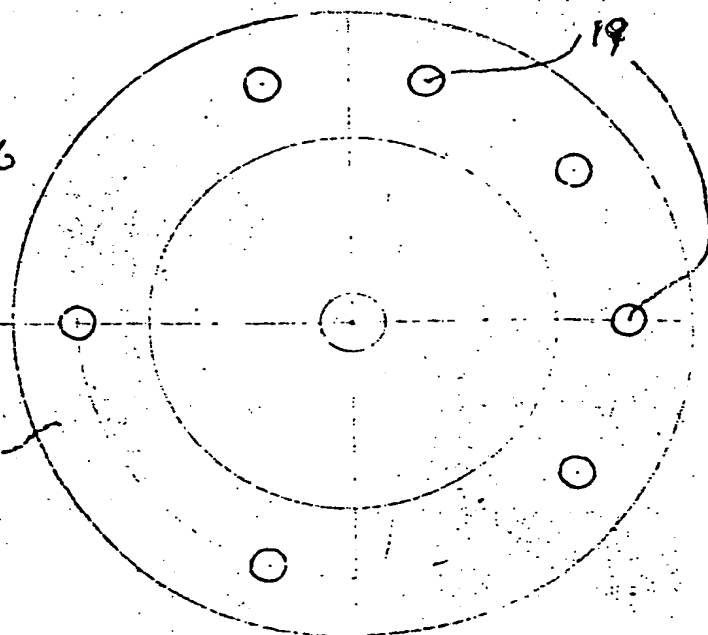


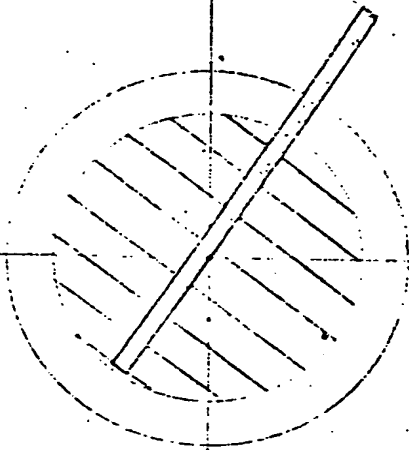
FIG 1B

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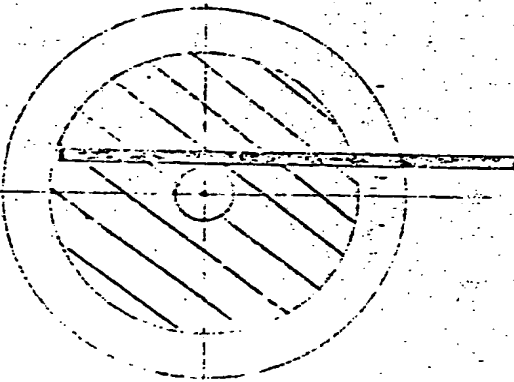
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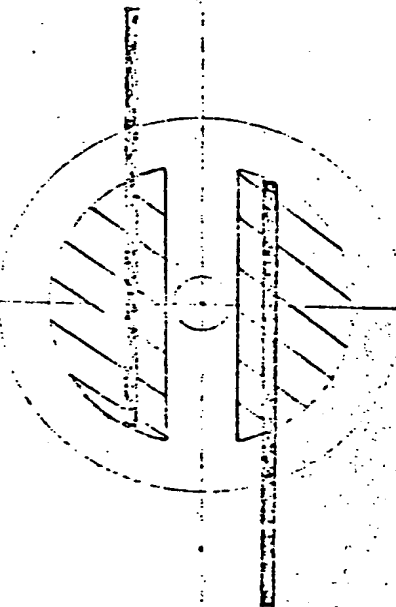
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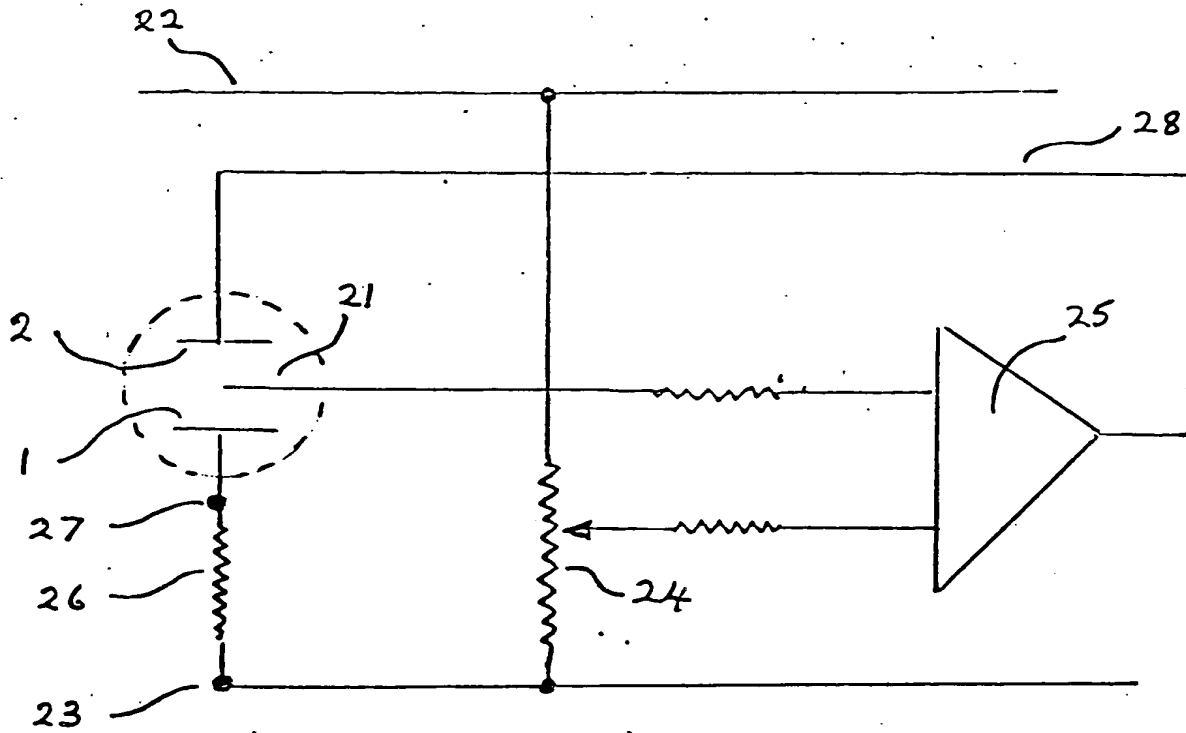


FIG 3

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